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Technical Report 56

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**Mechanics of Crack Growth
in Epoxide Adhesives**

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May 1978

20061102506

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PROPELLANTS, EXPLOSIVES AND ROCKET MOTOR ESTABLISHMENT
WALTHAM ABBEY, ESSEX

Technical Report No 56

Approved 24 8 77

MECHANICS OF CRACK GROWTH IN EPOXIDE ADHESIVES

by

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SUMMARY

This report considers the failure of structural adhesive joints consisting of metallic substrates bonded with various simple epoxide adhesives. The different modes of crack growth behaviour are discussed and possible controlling mechanisms considered.

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CONTENTS

	<u>Page No</u>
1 INTRODUCTION	5
2 EXPERIMENTAL	5
3 CRACK GROWTH BEHAVIOUR	7
3.1 Experimental Observations	7
3.2 Controlling Mechanisms	9
3.3 Morphological Considerations	13
4 CONCLUSIONS	14
5 ACKNOWLEDGEMENTS	15
6 REFERENCES	16
Figures 1 to 6	

1 INTRODUCTION

The term "structural" is commonly applied to a wide range of adhesives which are thermosetting, possess a high modulus and are used in load-bearing structures. Structural adhesive joints fail by progressive crack growth and thus no failure criterion based on average stress or modulus (reflecting, for example, gross yielding or buckling) is appropriate, and the failure criterion must be founded upon the initiation and propagation of flaws inherent in the joint. Since the basic tenet of continuum fracture mechanics theory is that the strength of most real solids is governed by the presence of flaws, and since the theory enables the manner in which they propagate under stress to be analysed mathematically, the application of fracture mechanics to adhesive joint failure has recently received considerable attention.

This report considers some of the different modes of crack growth that are observed in structural adhesive joints since this is of importance when interpreting, and designing with, the fracture mechanics data that are currently being collected on structural adhesives.

2 EXPERIMENTAL

The specimen geometry employed in this investigation was a tapered-double-cantilever-beam joint and is shown schematically in Figure 1.

The substrate material was either aluminium alloy, to specification British Standard 1474 NE4 or mild-steel, to specification British Standard 970 EN3B. The metal substrate was machined into cantilever beams 308 mm long, 12.7 mm thick and with a height, h , varying between 16.0 and 47.8 mm. The surfaces to be bonded were first subjected to a liquid- and vapour-degreasing bath of trichloroethane, then grit-blasted with 180-220 mesh alumina, then degreased again and finally allowed to air-dry. The epoxide adhesives employed were based upon a diglycidyl ether of bisphenol A (DGEBA resin) and were formulated as shown in Table 1. Immediately prior to joint preparation the metal substrates were treated as described above, adhesive spread on the treated faces and the two beams pressed lightly together; small pieces of plastic sheet, previously inserted in the adhesive at the far ends of the joint, were employed to control the thickness of the epoxide resin layer to 0.50 ± 0.06 mm. Further, a piece of Teflon tape, about 30 mm long, 6.4 mm wide and 0.08 mm thick, was previously placed approximately in the centre of the adhesive layer and at the narrow end of the joint, to assist in propagating a "starter" crack layer.

TABLE 1

Adhesives Employed and their Physical Properties

Epoxy resin	DGEBA	DGEBA	DGEBA
Curing agent	triethylene tetramine (TETA) (a primary amine)	tetraethylene pentamine (TEPA) (a primary amine)	tri-2-ethylhexanoate of 2,4,6-tris (dimethylamino-methyl) phenol (a tertiary amine)
Epoxy resin	11.0	10.0	9.4
Parts per hundred of resin used	2 h/23°C + 2 h/60°C	23 h/23°C + 8 h/80°C	96 h/23°C + 1½ h/100°C + 2½ h/180°C
Cure schedule	2.8	4.3	2.8
Youngs Modulus (E_a) (GPa) (a)	39.5	59.0	52.9
Breaking stress (MPa) (a)	1.6	1.7	3.7
Strain at break (%) (a)	81	98	85
Glass transition (°C) (b)	-15	-18	-17
β -relaxation temperature (°C) (b)	419	336	394
Average molecular weight between crosslinks (c)			

Notes: (a) Uniaxial tensile data; 23°C, 56% rh, strain rate = 0.0014 s^{-1}

(b) Determined from differential scanning calorimetry at a heating rate of 0.27°C/s .

(c) Calculated using the equation proposed by Nielsen.¹

The adhesive was cured as indicated in Table 1 and the specimens were then conditioned at 23°C and 56% rh, for a few days prior to testing.

To obtain a natural starter crack for subsequent experiments, the arms of the specimen were separated at a constant rate of 8.5×10^{-3} mm/s, using an Instron tensile testing machine, until the crack was about 70 to 100 mm long. The specimens were then re-loaded at various constant rates of displacement at 23°C and 56% rh until crack propagation was observed.

The adhesive fracture energy, G_{Ic} , was ascertained from the relationship:²

$$G_{Ic} = \frac{4 P_c^2 m}{E_s b^2} \quad (1)$$

where P_c is the applied load, E_s is the modulus of the substrate, b is the specimen thickness and m is a geometry factor given by:³

$$m = \frac{3c^2}{h^3} + \frac{1}{h} \quad (2)$$

where c is the crack length corresponding to a height of substrate beam, h .

The initial crack velocity was ascertained either by visual observation or, for crack velocities greater than about 0.01 m/s, by the crack progressively rupturing a conductive grid painted on the side of the specimen and recording the associated voltage change on an oscilloscope.

3 CRACK GROWTH BEHAVIOUR

3.1 Experimental Observations

Now we and other workers (eg Refs 4,5,6,7) have found that, upon loading structural adhesive joints at a constant rate of displacement in a hard testing machine, crack growth may occur in one of two basic modes:

- (i) In a continuous, steady manner at a constant load with the rate of crack growth being dependent upon the rate of displacement employed. This is termed "stable" crack growth and a typical load-displacement trace for a joint exhibiting this type of behaviour is shown in Figure 2a.

- (ii) Alternatively, crack propagation may occur intermittently in a stick-slip manner and load values appropriate to both crack initiation and crack arrest ascertained. This is termed "unstable" crack growth and a typical load-displacement trace is shown in Figure 2b.

These two different modes of crack growth are illustrated in Figure 3 where the adhesive fracture energy, G_{Ic} , is plotted as a function of the initial crack velocity, \dot{c} , for various joints. There are several noteworthy features.

First, for all types of joint the locus of joint failure was cohesive in the epoxide resin layer and, indeed, the crack propagated along the centre of the adhesive layer.

Second, for the joints prepared with the primary (1°) amine curing agents (TETA and TEPA), the crack propagated in a stable mode and crack velocities within the range 10^{-4} to 5 m/s were observed. The actual velocity attained was directly proportional to the rate of displacement of the specimen loading points (which, in turn, was determined by the crosshead speed of the testing machine) and this is shown in Figure 4. A velocity of 5 m/s represented the fastest crosshead speed that could be attained; higher crack velocities in these joints might well be achieved with higher crosshead speeds. Thirdly, although the joints prepared with the TETA-cured adhesive are marginally tougher, the value of G_{Ic} was virtually independent of crack velocity for both these 1° amino-cured adhesives. Also, both the mode of crack growth and the $G_{Ic} - \dot{c}$ relation were independent of the substrate material employed.

However, if instead of a 1° amine curing agent the tertiary (3°) amine curing agent was used to crosslink the epoxy adhesive only unstable crack growth was observed and the initial velocity of crack propagation was always greater than about 20 m/s. Velocities less than this were never recorded, no matter how slowly the arms of the specimen were separated; indeed there was no apparent correlation between crack velocity and the crosshead speed of the testing machine whatsoever. For the unstable cracks values of G_{Ic} for crack initiation and crack arrest were ascertained: values of G_{Ic} (initiation) rose rapidly with increasing crack velocity,

values of G_{Ic} (arrest)* were independent of initial crack velocity.

3.2 Controlling Mechanisms

The different epoxide adhesives so far discussed possess similar uniaxial tensile properties, glass transition and β -relaxation temperatures and average molecular-weights between crosslinks. (Table 1.) There are no obvious parameters to distinguish between these materials which, nevertheless, exhibit such different modes of crack growth behaviour.

3.2.1 Previous Work

It is instructive, therefore, to consider previous suggestions which have been advanced to explain stable versus unstable crack growth, not only in structural adhesive joints but also in other materials. These are (i) the effect of adhesive layer thickness, (ii) the fracture toughness decreasing with increasing crack velocity at some point in the toughness-crack velocity relationship and (iii) the geometry of the test specimen employed to determine the toughness-crack velocity data.

*The values of G_{Ic} (arrest) were calculated using a static analysis as embodied in Equations 1 and 2. Crack propagation and arrest currently represent a controversial topic in fracture mechanics. As Rosenfield and Kanninen⁸ point out, two incompatible viewpoints exist. One is that when arrest values are calculated from static analyses the process of crack arrest is being treated as the reverse in time of crack initiation and hence G_{Ic} (arrest) is a unique property of the material. The second viewpoint is that dynamic effects (stress waves, structural vibrations) and their possible influence on the process of crack arrest must be considered so that, as the crack accelerates in the initial phase, kinetic energy is built up in the specimen which is subsequently available to contribute to the crack driving force during the arresting phase. Kalthoff et al⁹ have presented experimental evidence that for fast propagating cracks, in parallel-double-cantilever-beam specimens of epoxide materials, dynamic effects have an influence on the crack arrest process, as predicted by the concept of recovered kinetic energy. These points should be borne in mind when considering the static G_{Ic} (arrest) values shown in Figure 3.

First, Bascom et al^{7,10} have elegantly demonstrated that for rubber-modified epoxide adhesives the value of G_{Ic} and the mode of crack growth are dependent upon the thickness of adhesive layer used. They showed that a transition from unstable to stable crack growth may occur when the thickness of the adhesive is reduced to slightly less than the size of the deformation zone at the tip of the crack. The radius of the plastic zone, r_{Iyc} , at the crack tip may be deduced from Equation 3.

$$r_{Iyc} = \frac{1}{6\pi(1 - \nu^2)e_y^2} \cdot \frac{G_{Ic}}{E_a} \quad (3)$$

Values of r_{Iyc} for the adhesives employed in the current work were therefore calculated using the data in Table 1 and Figure 3. A value of Poisson's ratio, ν , of 0.35 was assumed and values of the "yield strains", e_y , of about 1 to 2% taken (these values of e_y must be approximate since none of the adhesives exhibit bulk yielding in uniaxial tension). The sizes of the deformation zones, $2r_{Iyc}$, for the adhesives lie between about 14 to 70 μm and are, therefore, all considerably less than the thickness of the adhesive layer, which is 500 μm . Thus a plastic zone size/adhesive layer thickness effect cannot explain the different modes of crack growth observed in the present work.

Second, in poly(methylmethacrylate)^{11,12} slow, stable crack growth occurs at room-temperature giving a gently rising fracture toughness-crack velocity curve until, at a velocity of about 10^{-1} m/s, the fracture toughness falls rapidly with further increases in the crack velocity. This causes unstable crack growth to occur until, at still faster velocities, the fracture energy begins to rise steeply due to kinetic energy effects. The maximum in the fracture toughness-crack velocity relation for p.m.m.a. has been ascribed to a β -relaxation transition occurring¹¹ or to an isotherm/adiabatic transition at the crack tip which causes local softening and a reduction in toughness.^{12,13,14} This decrease in G_{Ic} with increasing crack velocity has also been proposed as the cause of unstable crack growth in metal/epoxide joints.^{7,15} However, none of the joints which exhibit unstable crack growth have ever been observed to show (a) any stable crack growth at all or (b) a maximum in the $G_{Ic} - \dot{c}$ relation (and hence a negative

value of $dG_{Ic}/d\dot{c}$), either in this previous work or in the current work.*

Thirdly, several authors^{14,16,17,18} have drawn attention to the fact that crack stability also depends upon the actual test geometry employed. Mai and co-workers^{17,19,20} have shown that, as far as crack stability is concerned, the tapered-double-cantilever-beam specimen is an inferior test geometry to the double-cantilever-beam specimen with parallel arms. Further, as the taper angle of the former test piece is increased the likelihood of obtaining stable crack growth diminishes. However, they have reported that crack stability may be markedly increased simply by turning the conventional tapered-double-cantilever-beam specimen around and applying the load at the thick end. We therefore prepared such joints consisting of aluminium alloy substrates bonded with the DGEBA/tertiary amine adhesive (which previously had exhibited only unstable crack growth). The joints were then loaded via the thick end at various constant rates of displacement but this different loading technique appeared to have no effect on the mode of crack growth; as before only unstable crack growth was recorded.

Thus, none of these above explanations accounts for the different modes of crack growth observed in the present studies.

3.2.2 Proposed Model

Considering the above, our own observations on the shape and sharpness of crack tips in the adhesives and comments of other workers,^{21,22,23} we suggest that the main parameter controlling the crack growth behaviour in our structural adhesive joints is simply the amount of localised plastic deformation that occurs close to the crack tip, prior to crack propagation. If the degree of plastic deformation is high the crack tip will be severely blunted. Hence, when the crack eventually propagates, the rate of release of stored energy will be greater than required for a stable moving crack, so the crack will rapidly accelerate, ie unstable crack growth results. Alternatively, if the extent of plastic deformation is more limited (ie the associated high strains cannot be accommodated at the crack tip prior to propagation), then

*This does not mean that the joints which show stable crack growth will necessarily always do so. If $dG_{Ic}/d\dot{c}$ is ever sufficiently negative a transition to unstable crack growth may well occur.

the blunting effect will be small and stable crack growth may result.

The localised plastic deformation at the crack tip results from either of two types of yield process: yielding in shear, and yielding under normal stresses. The latter is a dilatational process commonly referred to as craze formation and involves cavitation followed by plastic deformation of the cavities to give an orientated foam-like structure in limited regions of the material. The diameter of the pores or channels in a craze is typically a few tens of nanometres and the void content is about 40 to 60%. However, while there is definite proof for craze formation in rubber-modified epoxide materials,²⁴ the evidence for craze formation in simple epoxide materials at room temperature is less positive²⁵ and no evidence for craze growth was found in the present studies.

Now, as was previously mentioned, none of the adhesives showed bulk yielding in uniaxial tensile tests and no yield-load drop was observed. This is due to the adhesives' brittle fracture stresses being lower than their yield stresses under uniaxial tensile conditions. Therefore, to obtain information on their yielding behaviour and, in particular, on their ability to undergo plastic deformation, uniaxial compression tests were conducted.²⁶

True-stress/nominal-strain uniaxial compression curves (calculated assuming deformation occurred at constant volume) for the three adhesives at two strain rates* are shown in Figures 5 and 6. It is immediately evident

*A problem arises here since it is necessary to ascertain a strain-rate, \dot{e}_{tip} , appropriate to a volume element adjacent to the crack tip in order to relate the fracture results to this compression data. If t is the time scale of the deformation of the volume element and e_{tip} is the strain it undergoes, then

$$\dot{e}_{tip} \approx \frac{e_{tip}}{t} \quad (4)$$

If e_{tip} is taken as the maximum strain from the compression data and t as the time between the start of the fracture test and the instant of crack propagation (ie values of t between about 1 and 250 s), then \dot{e}_{tip} values will be approximately in the range 10^{-1} to 10^{-5} s^{-1} . These values are in a comparable range to the strain rates employed to determine the compression data.

that the DGEBA/tertiary-amine adhesive is capable of undergoing considerable plastic deformation before fracture. It will be recalled that joints prepared using this adhesive exhibited only unstable crack growth. In distinct contrast, the two primary-amine cured adhesives only withstood relatively low strains before they failed by pieces shattering off the specimen; joints prepared with these adhesives always fractured by stable crack propagation.

This model for explaining the crack growth behaviour in structural adhesive joints is also substantiated by results of other workers. For example, Young and Beaumont²⁷ found that unstable crack growth occurred in certain epoxide materials containing low volume fractions (0 and 0.15) of silica particles but stable crack propagation was observed when higher volume fractions of silica were incorporated. Uniaxial compression tests demonstrated that the unfilled and low-filler content materials were able to flow to over 30% strain before failure. However, this ability to undergo plastic flow decreased as the silica volume fraction was raised until, at a volume fraction of 0.52, the maximum plastic strain was about 12%.

Further, this model may explain observations that some epoxide materials in which stable crack growth normally occurs may, under certain circumstances, change their mode of cracking and exhibit unstable crack propagation. Such changes have been caused by (a) lowering the crosshead speed of the testing machine²³ or (b) by exposing the materials to water, or high humidity, for short periods prior to testing^{23,28} or (c) by applying a constant load just below the short-term fracture load for a few days prior to testing.²⁹ All these experiments would be predicted to increase the ability of the material to undergo greater deformation; due to plasticisation at the crack tip in (b) or to a lowering of $\dot{\epsilon}_{tip}$ in (a) and (c). Hence, in the vicinity of the crack tip more localised plasticity will occur, the crack will blunt to a greater degree and the possibility of unstable crack growth will be increased.

3.3 Morphological Considerations

We have demonstrated that the epoxide adhesives shown in Table 1 all possess high crosslink densities but are capable of undergoing markedly different amounts of plastic flow and this can be related to the mode of crack growth. This raises several interesting points. Namely, why should

these materials show such different plastic-deformation capabilities and why should one of them (ie the DGEBA/tertiary-amine cured adhesive) be able to accommodate such high plastic strains?

It is possible that the basic reasons for these observations are related to the morphologies of the materials. The morphology of thermosetting resins has recently generated considerable interest and several authors^{30,31} have suggested that epoxide materials contain crosslink density inhomogeneities of 10 to 60 nm in diameter. Labana et al³⁰ have presented a mechanism for crosslinking which predicts the formation of those spherical nodules or "gel balls", long before the gel point of the mixture is reached. The excessive intramolecular reactions within the gel balls exhausts the amount of available crosslinking agent, causing a deficiency in crosslink density in the regions between the gel balls.

The morphologies of the adhesives used in the present work have been studied by Aspbury et al.³² These workers obtained microtomed, carbon/platinum-shadowed sections of the adhesives and examined them in the transmission electron microscope at high magnifications. Nodular structures were observed and from microdensitometer tracings of the micrographs the median diameter of the nodules in the 3^o amine cured adhesive (which exhibited high plastic strain capability and gave unstable crack growth) was 41 nm. However, in the TETA- and TEPA-cured adhesives (which exhibited relatively low plastic strain capability and gave stable crack growth) the nodules were much smaller having median diameters of 28 and 24 nm respectively.

It is suggested, therefore, that it is the flow of nodules past one another, rather than the slippage of polymer chain segments, which is the mechanism involved and which is largely responsible for the plastic strains observed. Further, that the different plastic strains recorded may possibly be ascribed to morphological differences.

4 CONCLUSIONS

4.1 Experiments have been conducted employing tapered-double-cantilever-beam joints bonded with different epoxide adhesives. Depending upon the particular adhesive used, crack propagation occurred either (a) in a continuous stable manner with crack propagation velocities in the range 10^{-4} to 5 m/s or

(b) intermittently in an unstable manner when the initial crack velocity was never less than about 20 m/s and, in some instances, rose to about 450 m/s. Further, in (a) the adhesive fracture energy, G_{Ic} , was virtually independent of the associated crack velocity while in (b) value of G_{Ic} for crack initiation increased rapidly with increasing velocity and values of G_{Ic} for crack arrest were independent of initial crack velocity.

4.2 Mechanisms for these different modes of crack growth have been considered. It is proposed that the amount of localised plastic deformation, arising from shear yielding, that occurs at the crack tip prior to crack propagation is the controlling feature. Correlations between the mode of crack growth, ability to undergo plastic straining and morphology of the adhesive have been established in support of the proposed mechanism.

5 ACKNOWLEDGEMENTS

The authors would like to thank Mrs M Cortline, and Mr P Maher for their assistance with various aspects of the experimental work. Also, Mr W A Dukes and Dr A K Green for helpful criticisms and suggestions and Mr P Aspbury, Mr K W Allen and Professor W C Wake of City University for their cooperation and permission to publish data from their morphological studies.

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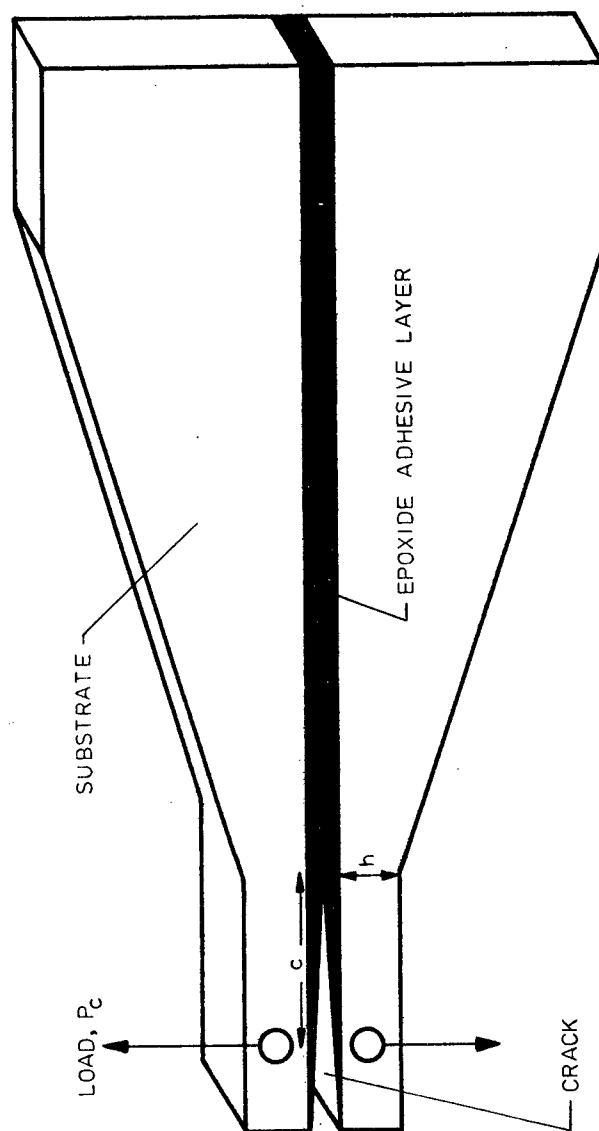


FIG. 1 SKETCH OF TAPERED-DOUBLE-CANTILEVER-BEAM SPECIMEN

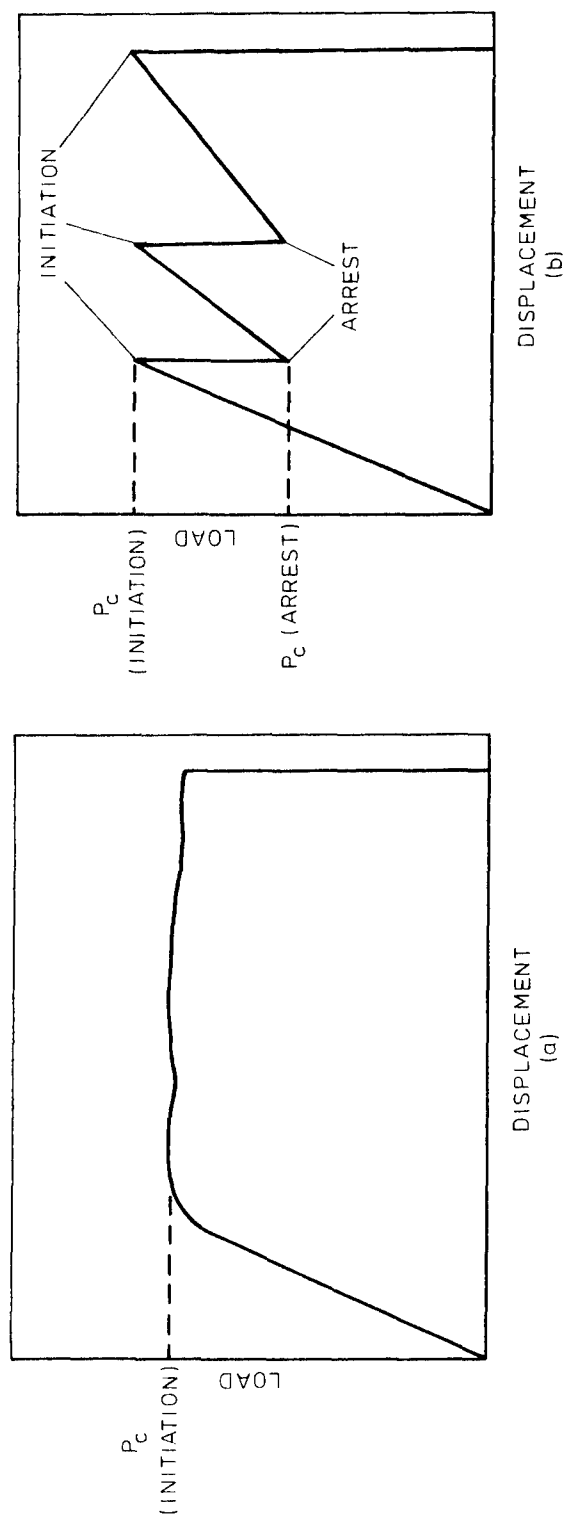


FIG. 2 (a) STABLE CRACK GROWTH (b) UNSTABLE CRACK GROWTH

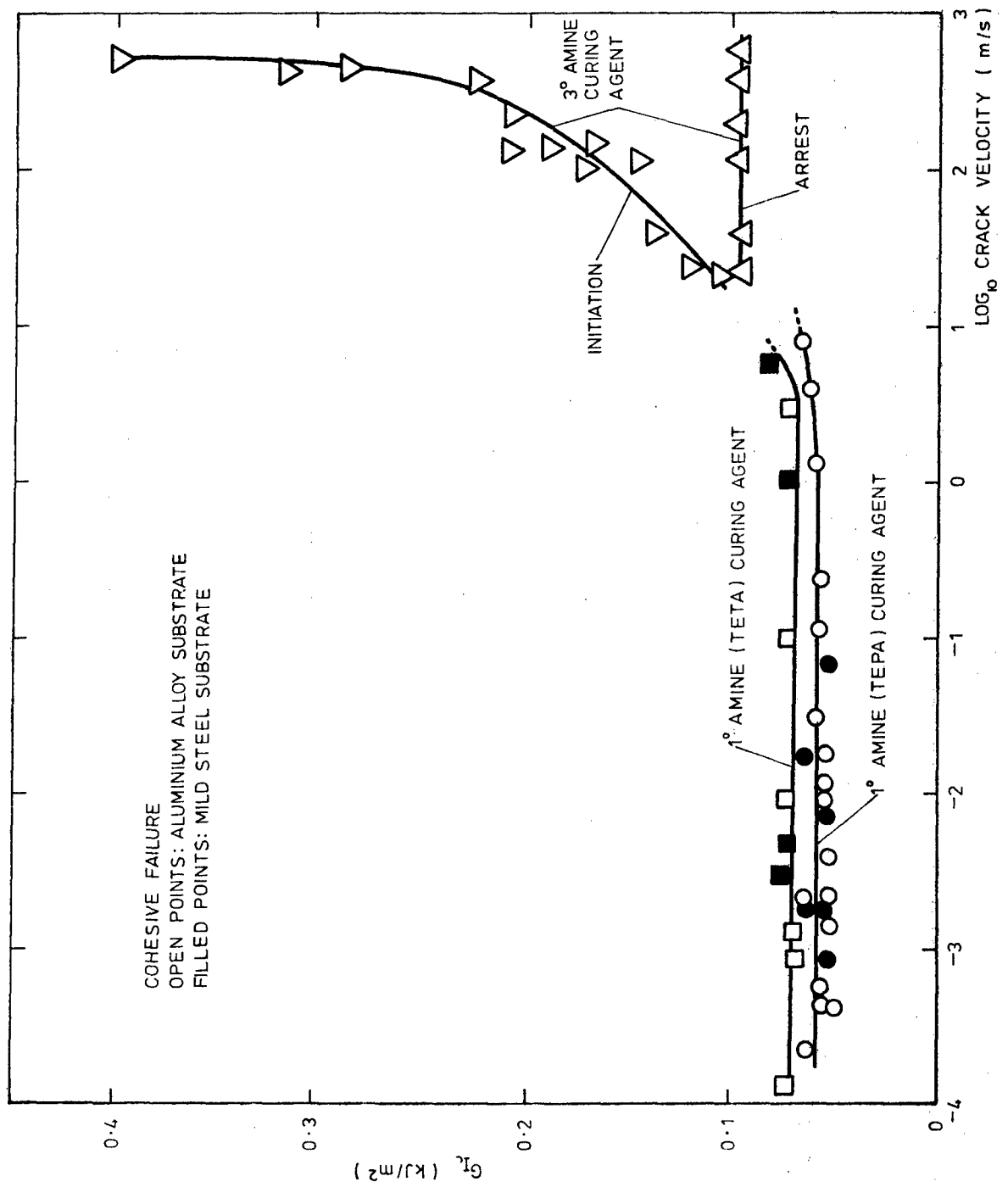


FIG. 3 ADHESIVE FRACTURE ENERGY, G_{Ic} , VERSUS CRACK VELOCITY

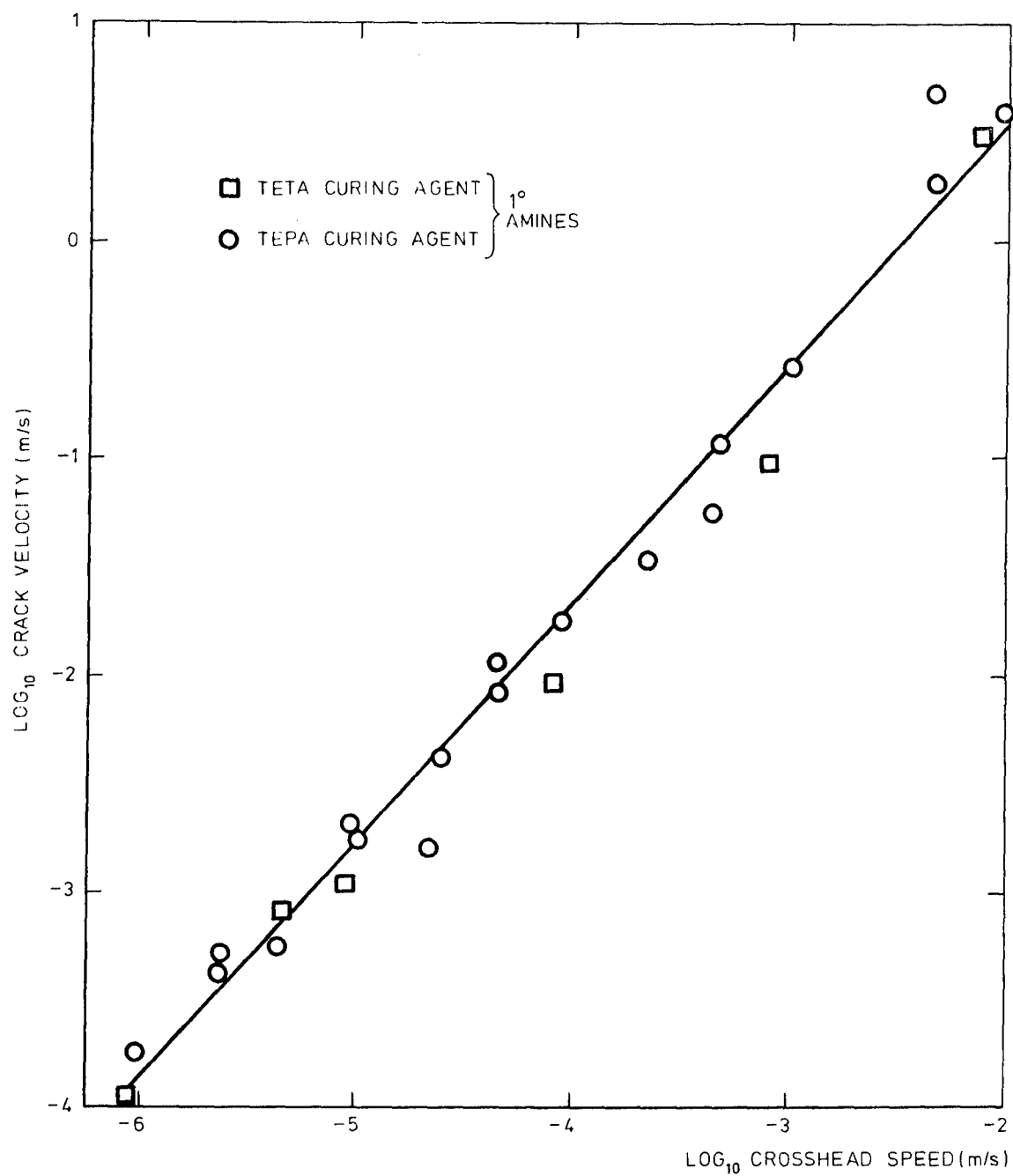


FIG. 4 CRACK VELOCITY AS A FUNCTION OF CROSSHEAD SPEED

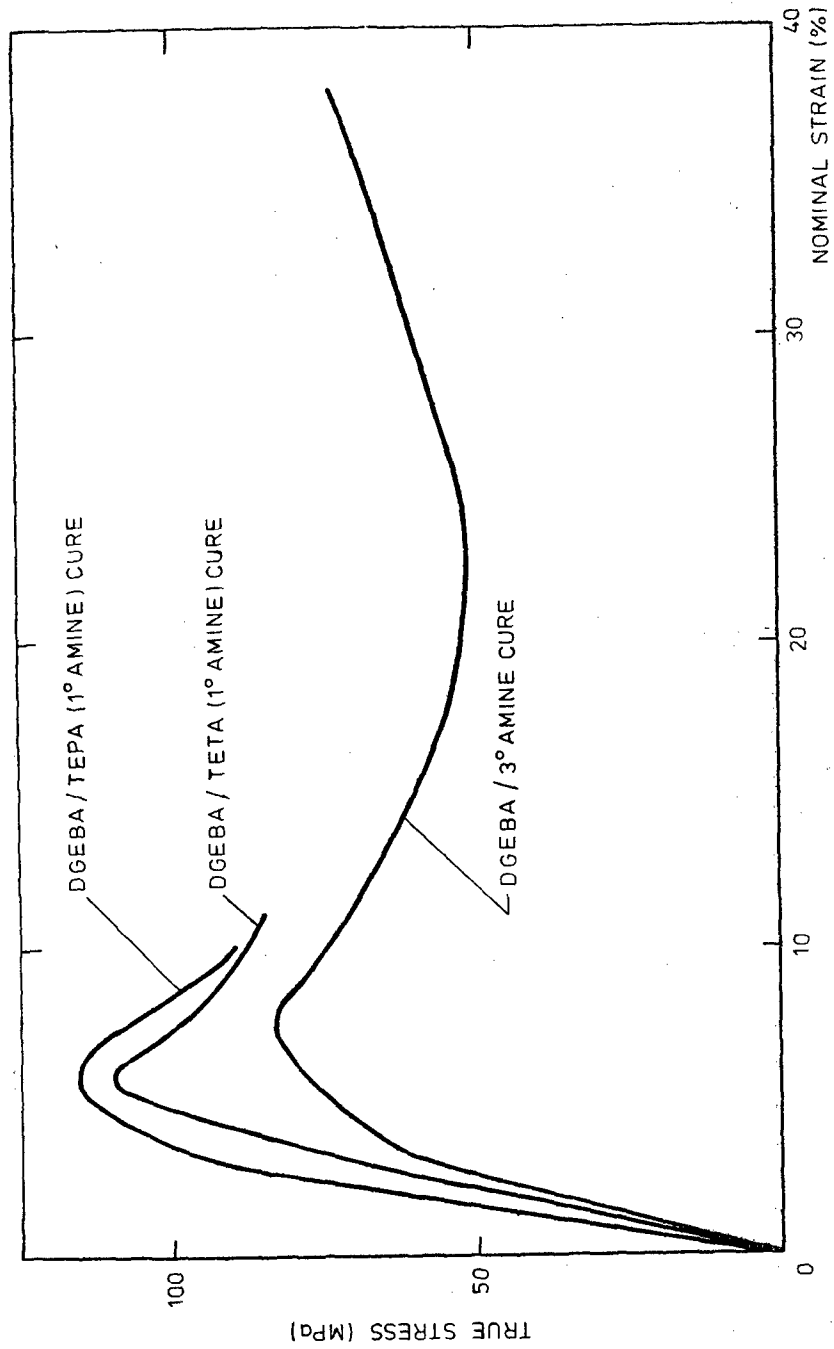


FIG. 5 UNIAXIAL COMPRESSION CURVES FOR THE EPOXIDE ADHESIVES
($\dot{\epsilon} = 6 \times 10^{-2} \text{ s}^{-1}$)

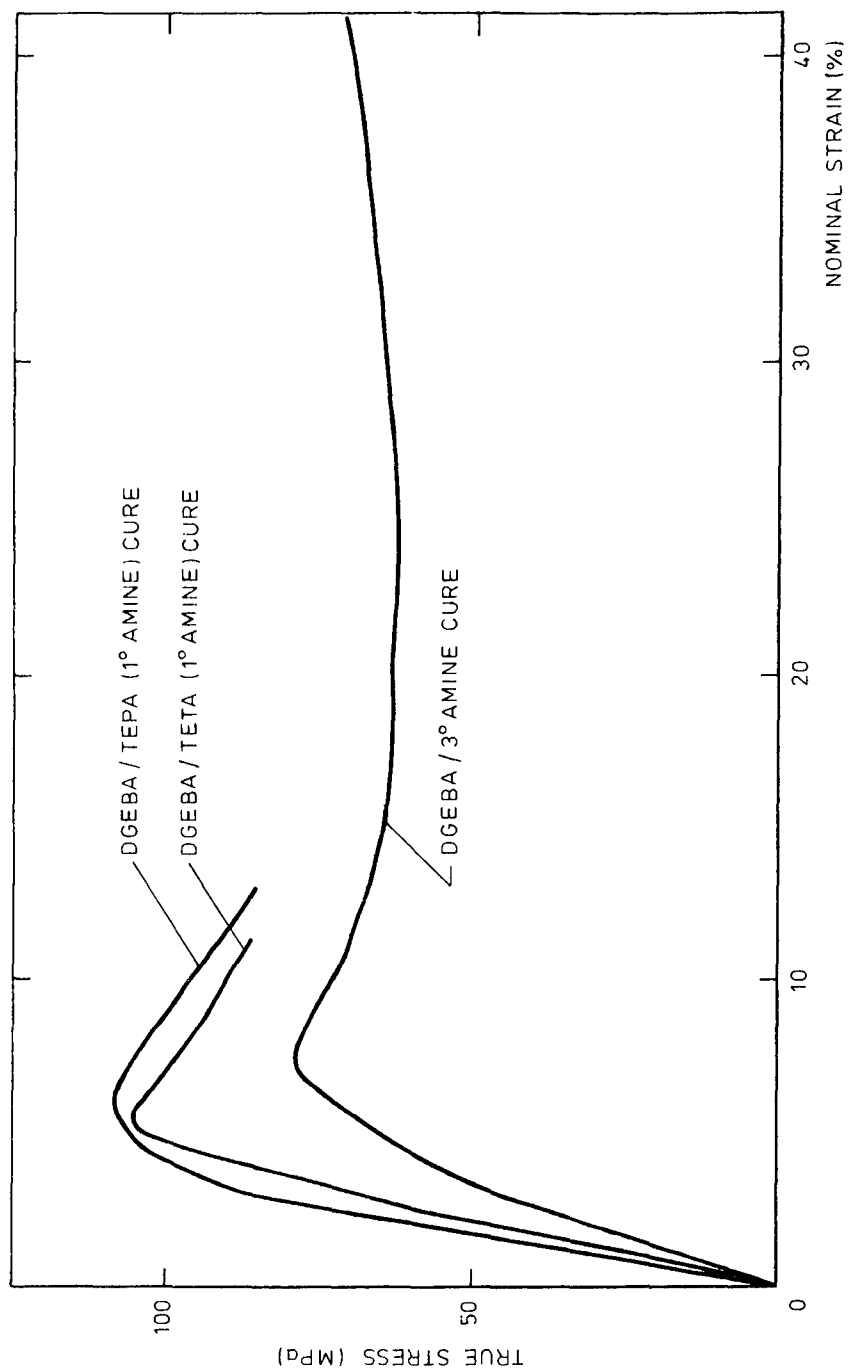


FIG. 6 UNIAXIAL COMPRESSION CURVES FOR THE EPOXIDE ADHESIVES
($\dot{\epsilon} = 6 \times 10^{-4} \text{ s}^{-1}$)

REPORT DOCUMENTATION PAGE

(Notes on completion overleaf)

Overall security classification of sheet **Unlimited**

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1. DRIC Reference (if known)	2. Originator's Reference PERME TR 56	3. Agency Reference	4. Report Security Classification UNLIMITED
5. Originator's Code (if known) 7281400E	6. Originator (Corporate Author) Name and Location Propellants, Explosives and Rocket Motor Establishment Waltham Abbey Essex, England		
5a. Sponsoring Agency's Code (if known)	6a. Sponsoring Agency (Contract Authority) Name and Location		
7. Title MECHANICS OF CRACK GROWTH IN EPOXIDE ADHESIVES			
7a. Title in Foreign Language (in the case of translations)			
7b. Presented at (for conference papers). Title, place and date of conference			
8. Author 1. Surname, initials Gledhill R A	9a. Author 2 Kinloch A J	9b. Authors 3, 4...	10. Date pp ref 5.1978 19 32
11. Contract Number	12. Period	13. Project	14. Other References
15. Distribution statement			
Descriptors (or keywords) Adhesive bonding, Adhesives, Crack propagation, Cracking (fracturing), Morphology, Substrates, Aluminium alloys, Steels, Epoxy resins, Mechanical properties <div style="text-align: right;">(TEST)</div>			
Abstract This report considers the failure of structural adhesive joints consisting of metallic substrates bonded with various simple epoxide adhesives. The different modes of crack growth behaviour are discussed and possible controlling mechanisms considered.			